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16. ABSTRACT

Ambient air was sampled using distributed air volume sets of tandem Tenax beds at several locations. Apparent concentrations at some sites were ill behaved. Significant amounts of materials sometimes appeared on back beds where that would not be expected from retention volume estimates. Contamination and operating errors cannot explain the observations. Unidentified chemical reactions during sampling and/or thermal desorption are implicated. Reactions observed or reported in laboratory situations during simulated sampling or thermal desorption yield products of the kinds observed in these atmospheric samples.

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APPARENT REACTION PRODUCTS DESORBED FROM TENAX USED TO SAMPLE AMBIENT AIR

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Abstract—Ambient air was sampled using distributed air volume sets of tandem Tenax beds. *In situ* estimates of retention volumes were made and sometimes found to differ greatly from literature values. Inconsistent sets of apparent concentrations not due to contamination, operating errors or blanks were often observed. Unidentified chemical reactions during sampling and/or thermal desorption are implicated. It is concluded that empirical evaluation of data from Tenax sampling of ambient air is warranted in each sampling situation.

Key word index: Tenax sampling, air sampling, trace organic sampling, Tenax artifacts.

INTRODUCTION

Solid adsorbents are commonly used to selectively concentrate air contaminants. Tenax continues to be studied for an expanded range of applications (Billings and Bidleman, 1983; Pankow and Kristensen, 1983). Recently, laboratory studies of chemical reactions dependent on the surface condition, adsorbed species and the presence of oxidants have been reported (Roberts *et al.*, 1984; Pellizzari *et al.*, 1984). These studies neither suggest how to enumerate reaction possibilities nor how to identify and correct for their presence in applications, yet artifact reactions frustrate the straightforward use of analytical results to obtain atmospheric concentrations.

An empirical sampling approach which can reveal the presence of complications has been suggested (Walling, 1984). This approach, called distributed air volume sampling, requires simultaneous collection of several samples, each at a very different flow rate. Many interfering processes scale nonlinearly with volume sampled. If they are important, apparent atmospheric concentrations become dependent on the volume sampled. For example, benzaldehyde, an artifact from the reaction of Tenax and ozone (Roberts *et al.*, 1984; Pellizzari *et al.*, 1984), yields inconsistent concentrations in distributed air volume sets (Walling, 1984).

This sampling approach will not permit identification or enumeration of any interferences. However, the inclusion of tandem beds, i.e. two beds in series, and/or presampling spikes of deuterated species can assist in identifying adsorbate loss through inadequate retention. If losses, contamination and operating errors are absent, chemical reactions remain as the only explanation of inconsistent concentrations.

To assess the effects of artifact reactions on data from air sampling, distributed air volume sets of tandem Tenax beds were collected. Some contained presampling spikes of deuterated species. Reactions during thermal desorption of spiked Tenax were also observed to assess the possible importance of that little studied step.

EXPERIMENTAL

Materials

As described elsewhere (Bumgarner, 1981), Tenax-GC of differing and uncontrolled histories was solvent cleaned, desorbed and packed in cartridges containing 1.2-g beds. Cartridges were placed in individual culture tubes with Teflon-lined caps and stored under refrigeration except during shipment and sample collection.

Fifty-seven organic compounds, principally aromatic and halogenated hydrocarbons, were obtained in the highest purities commercially available and checked chromatographically. Generally, they were found to be more than 99% pure and were used as received.

Sampling

The samplers used have been described in detail elsewhere (Walling and Bond, 1984). Flows were regulated with calibrated mass flow controllers. Special checks for leaks and flow reading errors were routine.

Culture tubes containing cartridges needed for a sampling set were put in a metal can sealed with a compression fit lid. That can was placed in a shipping container. All shipping was expedited. Elapsed times between final desorption and analysis was routinely under 30 days and most often a week.

Spiking

Gaseous mixtures containing standards were prepared in helium as described elsewhere (Morris *et al.*, 1983). For presampling spikes, aliquots were withdrawn by syringe and entrained in a stream of cleaned helium which passed through a mildly heated zone and into a Tenax sampling cartridge. Two liters of helium were used. Post sampling spikes of two internal standards used only for quality control were done by direct injection into the bed.

Analysis

Tenax was thermally desorbed and the eluent cryofocused on a 50 m × 0.5 mm id SE-30 WCOT glass capillary in a Finnigan 4021 GC/MS. The GC was temperature program-

med from 30 to 240°C with the spectrometer scanning from 35 to 320 m z^{-1}. Data were processed by an automated procedure featuring a reverse library search for specific compounds, stringent qualitative identification criteria and quantification using extracted ion current profile areas in quadratic calibration curves. Detection limits for substances reported here are between 1 and 3 ng. Complete details of the experimental procedures, data processing and quality control are available (Berkley *et al.*, 1984).

RESULTS AND DISCUSSION

Table 1 contains three sets of results from 2-h exposures at a single site. Only three unspiked tandem beds and one unspiked single bed were included in each set because of the need to analyze over 300 samples in a brief period. The results shown are typical of this site at that time in the sense of common concentration dependence on volume and substantial material on some back beds at small volumes. They are a cross-section of the concentrations observed.

The repeatability of the sampling and analytical system has been estimated (Walling, 1984). About 95% of the differences were smaller than 2 μg m^{-3} for benzene and methylbenzene and half of that for other compounds. For concentrations larger than 10 μg m^{-3}, differences of 20% and 10%, respectively, are appropriate. Therefore, any concentration difference within a set exceeding these values indicates some complication(s).

Inspection of Table 1 reveals that complications are the rule. Therefore, consideration of (a) operating errors, (b) contamination, (c) inadequate blank corrections, (d) inadequate retention and (e) chemical reactions is needed. In every case independent quality assurance evidence indicates no operating error in sampling or analysis. A substance not requiring a blank correction, often tetrachloroethene, displays consistent behavior in every set confirming this finding.

Table 1. Net apparent atmospheric concentration (μg m^{-3}) from three distributed air volume sets of three tandem and one single Tenax bed

Tube Position†	F	B	F	B	F	B	F	Blank
Air Volume, ℓ	6	6	11.4	11.4	26.9	26.9	54	(ng)
Substance	Date 07/4, Temp. 16–23°C							
Benzene	7.0	6.1	4.9	3.9	3.9	1.3	2.1	(9)
Methylbenzene	57.0	22.0	50.4	21.3	53.6	1.8	20.7	(10)
1,2-Dimethylbenzene	3.8	2.0	3.8	1.2	2.4		1.9	
Ethylbenzene	3.4	2.8	2.7	2.0	2.0	0.38	1.6	
Trichloroethene	0.65	0.86	0.44	0.69	0.39	0.10	0.22	
1,1,1-Trichloroethane	5.5	17.9	2.9	11.0	2.1	1.2	1.0	(14)
Tetrachloroethene	2.2		2.0	1.3	2.1	0.12	1.6	
Benzaldehyde	32.4	29.4	20.0	13.0	11.0	7.0	5.6	(25)
1-Phenylethanone	32.4	30.3	29.3	10.1	10.4	7.0	9.1	
Benzonitrile	2.9	2.7	1.7	1.2	1.0	0.55		
Substance	Date 08/4, Temp. 16–19°C							
Benzene	17.1	2.6	15.9	1.9	15.6	0.9	13.4	(13)
Methylbenzene	122	9.9	126	6.9	22.6	1.8	10.4	(30)
1,2-Dimethylbenzene	9.6		9.3		8.5		8.3	
Ethylbenzene	6.3	0.83	6.1	0.52	6.2	0.17	6.1	(8)
Ethenylbenzene	1.2	0.60	1.2	0.35	1.1	0.16	1.4	(7)
Trichloroethene	3.4		3.3	0.3	3.3		3.0	
1,1,1-Trichloroethane	8.6	1.2	8.8	3.4	6.5	2.4	4.2	(25)
Tetrachloroethene	38.4	0.92	35.8	0.49	34.9	0.23	35.1	
1,2-Dichloropropane	18.5		19.2		20.6		21.0	
Benzaldehyde	11.3	10.5	10.7	3.8	5.6	2.6	4.6	(36)
1-Phenylethanone	0.39	9.9	14.7	0.78		1.9	3.2	(17)
Benzonitrile		2.0				0.49		
Substance	Date 15/4, Temp. 19–20°C							
Benzene	4.6	3.9	3.7	1.4	2.8	0.78	2.5	(12)
Methylbenzene	7.9	5.5	9.4	1.3	8.5	0.94	6.7	(12)
1,2-Dimethylbenzene	2.6		2.4		1.6		1.3	
Ethylbenzene	1.0	0.58	1.1	0.19	1.0	0.11	0.9	(8)
Ethenylbenzene	0.75	2.5	0.85	0.20	0.41	0.47	0.23	(8)
Trichloroethene	0.39	0.68	0.37	0.40	0.26	0.13	0.17	
1,1,1-Trichloroethane	1.7	2.9	1.9	1.3	1.6	0.63	0.8	(11)
Tetrachloroethene	2.8		2.9	0.25	2.7		2.6	
1,2-Dichloropropane	2.9		3.2		3.6		3.7	
Benzaldehyde	27.7	19.4	16.3	8.1	6.1	5.0	4.0	(34)
1-Phenylethanone	34.4	14.3	*	7.3	6.8	2.9	6.0	
Benzonitrile	2.3	1.6	1.4	0.85		0.39		

†F is front tube of a tandem pair. B is backup tube of the pair.
*Not quantitated, instrument malfunctioned.

Substantial material on backup beds is common in these data and the larger group which these represent. Blank corrections are results from an extra cartridge shipped as a sample but not exposed. Therefore, there is no reason to suspect some inadequacy of the blank correction due to special handling or selection. Common contamination that never affects blanks handled in this manner is improbable. Therefore, blank inadequacy and contamination are rejected as explanations.

Retention depends on some factors which cannot be controlled, e.g. gas composition. However, a chromatographic model is useful to evaluate breakthrough. Laboratory evaluation of these beds using dilute single adsorbate mixtures yielded a ratio of outlet to inlet concentrations of about 0.15 when the volume sampled was 0.5 of the retention volume. The ratios were stable over the range of flows used here (Walling *et al.*, 1982).

A standard chromatographic model implies that the number of theoretical plates is more than two and less than eight (Senum, 1981). Two theoretical plates requires that the concentration ratio actually observed at 0.5 retention volume be seen at the very inception of sampling. Eight theoretical plates implies a ratio of about 0.1 at 0.5 retention volume; a value somewhat smaller than generally seen.

Assuming four theoretical plates, the model indicates a sampling efficiency of 95% at very small sampling volumes and 80% at about 0.9 of the retention volume. Therefore, if the largest volume sampled in a distributed air volume set is about 0.9 of the estimated retention volume, loss of material should be seen as monotonically decreasing concentration and a bias of roughly 15% between extreme volumes. That difference should be difficult to observe because it approaches the repeatability of the procedure.

With tandem beds, losses from the front beds should be seen as increasing concentrations on the backup beds so that the sums of the concentrations from the pairs are constant. Detectable decreases in these sums should begin at volumes larger than about 1.8 retention volumes. This model implies then, that given the repeatability of the procedure, inadequate retention should be difficult to observe unless the volume sampled is nearly the retention volume for single beds and twice that for tandem beds.

For these beds at 20°C, only three substances in Table 1 have estimated retention volumes less than 100 ℓ (50 for benzene and trichloroethene and 15 for 1,1,1-trichloroethane) (Brown and Purnell, 1979; Krost *et al.*, 1982). Using these values, the model indicates that all substances should have been adequately retained by the tandem beds through the 26.9 ℓ samples. Simple inspection shows that neither the invariability of the sums nor the pattern of distribution between beds is present. Only 1,2-dichloropropane and tetrachloroethene results are generally well behaved. Some losses cannot be ruled out but inadequate retention cannot be the entire explanation. Use of very different retention volumes will not alter that conclusion.

Inadequacy of the other explanations requires that chemical reactions be considered. In laboratory situations reactions of Tenax with strong oxidizing agents were reported to yield hydrocarbons, benzaldehyde and 1-phenylethanone (Roberts *et al.*, 1984; Pellizzari *et al.*, 1984). Other reaction paths may exist for the oxygen-containing compounds. Some blanks were retained in the laboratory and handled so that oxidants were excluded except for brief exposure to air during transfer between equipment. Analysis of these blanks yielded benzaldehyde and usually 1-phenylethanone. The usual desorption parameters were a helium flow of 10 ml min^{-1} for 8 min at 270°C. However, if a blank was desorbed at 20 ml min^{-1} for 20 min but at the same temperature, the amounts of these two materials increased by a factor of about 2.5. The quantity found is more linearly related to the time spent at elevated temperature than the helium volume. Furthermore, if a tube was used to sample air, analyzed, stored for a day as if it were a blank, and then reanalyzed, these two substances were again found in amounts equal to those from a normal blank. It seems that more can be generated without additional oxidants. These experiments suggest, but do not demonstrate conclusively, that the Tenax decomposes on heating under helium to yield benzaldehyde and 1-phenylethanone.

Reactions of adsorbates on Tenax with chlorine to produce halogenated species have been reported (Pellizzari *et al.*, 1984). However, reactions can also occur during desorption. To evaluate this, cleaned blanks were spiked with gaseous mixtures containing several compounds, including bromotrichloromethane and pentachloroethane, and then analyzed. Recoveries of these two substances, spiked in nearly equal amounts over a range from 100 to 500 ng, were uniquely and conspicuously poor. Tri- and tetrachloroethene were absent from all starting materials. However, trichloroethene was desorbed from the spiked Tenax. The amounts were approximately 20% of the spike, i.e. 20–100 ng. Tetrachloroethene also appeared in linearly increasing amounts up to 60 ng, but it did not increase with spikes larger than 300 ng. Although the poor haloalkane recoveries do not prove that they were reactants, the mass loss was in excess of the mass of the new haloalkenes observed.

Since reactions have been observed involving all classes of substances in Table 1, ascribing the peculiar behavior to reactions is reasonable. The effects are numerically significant but there are no data to facilitate corrections. It is impractical to make quantitative estimates of effects from distributed air volume sets. Therefore, such data cannot be used to arrive at an estimate of the contents of the air sampled.

The previous discussion contains two assumptions: (1) published retention volumes are applicable, and (2) large concentration increases were absent late in the sampling period. Retention volumes are functions of composition (Bertoni *et al.*, 1981; Walling *et al.*, 1982). Changes in composition during sampling cannot be observed, but an ambiguous indication of concen-

Table 2. Net apparent atmospheric concentrations ($\mu g\,m^{-3}$) and spike recoveries

Air volume (l)	Front 6	Backup 6	Front 11.4	Backup 11.4	Front 27.2	Backup 27.2	Front 54	Backup 54	Avg. blank (ng)	Avg.	S.D.
				Date: 23/1, Temp. −1 to +4°C							
Benzene	7.2	−2.20	7.9	−0.88	7.5	−0.43	8.5	−0.33	(39)	7.83	0.63
Methylbenzene	12.4	−1.30	12.1	0.34	13.4	−0.32	13.9	−0.33	(18)	12.98	0.92
1,2-Dimethylbenzene	3.73		3.20		3.28		3.47			3.42	0.25
1,3,5-Trimethylbenzene	0.78		0.78		1.11		1.31			1.00	0.28
Ethylbenzene	2.56	−0.67	2.33	−0.35	2.48	−0.15	2.77	−0.07	(4)	2.54	0.19
Ethenylbenzene	0.63	−0.36	0.56	−0.07	0.61	−0.22	0.52	−0.04	(6)	0.58	0.05
1,1,1-Trichloroethane	1.48				1.34		0.86	0.25			
Benzaldehyde	0.47	−0.73	1.62	−0.60	1.42	−0.03	1.87	−0.24	(71)		
1-Phenylethanone	0.40	−0.30	0.63	0.73	1.34	0.17	2.87	−0.20	(51)		
Recoveries*											
Benzene-d_6	0.94	0.00	0.95	0.00	0.97	0.00	1.03	0.00	1.00		
Methylbenzene-d_8	0.99	0.00	0.91	0.00	0.98	0.00	1.02	0.00	1.00		
Air volume (l)	Front 6	Backup 6	Front 11.4	Backup 11.4	Front 27.1	Backup 27.1	Front 54	Backup 54	Avg. blank (ng)	Avg.	S.D.
				Date: 27/4, Temp. 28–30°C							
Benzene	4.32	0.42	2.51	0.11	2.25	0.53	1.57	0.66	(27.5)	3.10	1.1
Methylbenzene	5.1	−0.75	4.81	−0.39	5.2	−0.17	4.85	−0.08	(4.5)	5.01	0.1
Ethylbenzene	1.67		1.39		1.36		1.21			1.41	0.9
Ethenylbenzene	0.79	0.27	0.30	0.17	0.19	−0.13	0.06	0.03	(3.5)		

1,3/1,4-Dimethylbenzene	3.83		3.99		4.45		4.17	0.39		4.11	0.7
Benzaldehyde	6.0	1.48	1.50	1.70	2.24	0.04	2.35	−0.79	(77.5)		
1-Phenylethanone	0.53	−9.1	3.88	1.18	3.47	−0.18	3.56	−1.02	(55)		
Benzonitrile	1.98					0.22					
Recoveries											
Benzene-d_6	1.06	0.00	0.94	0.04	0.81	0.23	0.19	0.73	0.94		
Methylbenzene-d_8	1.08	0.00	1.03	0.00	1.10	0.00	1.03	0.00	0.97		
Air volume (l)	Front 6	Backup 6	Front 11.4	Backup 11.4	Front 27	Backup 27	Front 54	Backup 54	Blank (ng)	Avg.	S.D.
					Date: 23/8, Temp. 25–30°C						
Benzene	5.5	−0.47	3.04	−0.86	3.47	−1.93	2.54	0.74	(52)		
Methylbenzene	8.4	−1.33	8.00	−0.70	8.3	−0.30	7.6	−0.15	(8)	8.11	0.38
1,3/1,4-Dimethylbenzene	6.5	0.20	6.30		6.9		5.5	0.02		6.30	0.62
1,3,5-Trimethylbenzene					0.44		0.55				
Ethylbenzene			1.87		1.90						
Ethenylbenzene	−0.67	0.70	−0.35	−0.35	0.36	−0.15	0.29	0.05	(4)		
1,1,1-Trichloroethane							0.42	0.42			
Tetrachloroethene					2.86		2.98			2.92	0.08
Tetrahydrofuran	0.34		0.15		0.10	0.06					
Benzaldehyde	6.2	7.1	3.14	−0.06	3.16	0.91	0.77	0.41	(109)		
1-Phenylethanone	7.2	18.6	8.7			0.91		0.66			
Benzonitrile	2.50	2.30		0.74		0.36		0.21			
Recoveries											
Benzene-d_6	0.72	0.43	0.40	0.70	0.00	1.08	0.00	0.69	1.10		
Methylbenzene-d_8	0.88	0.19	0.73	0.33	0.31	0.80	0.15	0.97	1.02		

*Recovery $= \frac{\text{ng found}}{226}$.

tration increases late in a period can be obtained if actual retention volumes are known. Therefore, it is desirable to attempt some *in situ* determinations of retention volumes.

Deuterated compounds were used for that purpose. Elution profiles of some normal and deuterated analogs from a spiked Tenax bed showed, as anticipated, consistent shapes and indistinguishable retention times. Assuming that such behavior could be expected of all species physically adsorbed on Tenax at ambient temperatures, a presampling spike of the labeled species was put on the front bed of a tandem pair to estimate retention volumes *in situ*. In a distributed air volume set the effective retention volume is between the two samples bracketing 50% recovery of the spike. However, good recovery of an isotopically altered species does not guarantee the absence of reactions since normal species might result from reactions of other normal adsorbates.

Table 2 contains results from distributed air volume sets of tandem cartridges collected over 2-h periods at another site. Prior to sampling, the front bed of each pair, i.e. the bed into which ambient air was drawn directly, was spiked with 226 ng each of benzene-d_6 and methylbenzene-d_8. Again, no evidence of operational errors was found.

Using literature values, no elution losses were anticipated for methylbenzene. Elution of benzene from the front bed was expected only on 27/4 and 23/8 since its retention volume at 29°C can be estimated to be about 30 ℓ (Krost *et al.*, 1982).

Total recoveries of the deuterated species from the tandem beds are excellent and other aspects are in agreement with the chromatographic model outlined above. However, on 23/8 the apparent retention volumes are $< 11.4\ \ell$ for benzene-d_6 and $< 27\ \ell$ for methylbenzene-d_8. Sporadic large reductions in retention volumes have also been observed elsewhere. Therefore, literature values may be applicable sometimes and seriously wrong others.

On 23/1 and 27/4 some negative concentrations of unlabeled species appear on backup beds. In some cases the masses they imply are relatively constant and essentially equal to the blank correction. Only blanks and front tubes were spiked. The correction is then applicable to the front tubes but slightly overcorrects the backups. Some unsuspected contamination of the spiking mixture may have occurred. In other cases the blank variability seems reasonable.

On 23/8 contamination might explain negative methylbenzene values but certainly not benzene's. The inconsistent behavior between the normal and deuterated species is the ambiguity suggesting the possibility of a concentration fluctuation. A brief end of period sampling of a large concentration increase might yield such results. Artifact chemical reactions which scaled linearly with sampling volume could do likewise. No resolution is possible from these data.

Average concentrations in Table 2 are presumptively applicable to the atmosphere sampled, but remaining data suggest the operation of chemical reactions. Data in both Tables suggest that chemical reactions are common when the atmosphere is sampled using Tenax. Data for substances affected by reactions cannot be used to estimate atmospheric concentrations because quantitative corrections are presently impractical.

CONCLUSIONS

Sampling of ambient air using Tenax is practical, but retention volumes sometimes differ greatly from literature values and chemical reactions during sampling and thermal desorption are common. Benzaldehyde, 1-phenylethanone and benzonitrile always displayed corrupted behavior. Use of Tenax to obtain atmospheric concentrations for these compounds is unlikely to succeed.

For aromatic and halogenated hydrocarbons inconsistent patterns of behavior have been observed. Therefore, for these classes, an empirical evaluation of data from each sampling situation using distributed air volume sets seems necessary. In the absence of that, customary assumptions could lead even to erroneous qualitative conclusions about the atmosphere sampled.

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